IS 4166 (1967): Methods of sampling of ilmenite and rutile [MTD 13: Ores and Raw Materials]

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IS : 4166 - 1967
(Reaffirmed 2005)

Indian Standard

METHODS FOR SAMPLING OF ILMENITE AND RUTILE

(First Reprint DECEMBER 1981)

UDC 549.641.23+549.514.63:620.113

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Rs 6·00

September 1967

Indian Standard

METHODS FOR SAMPLING OF ILMENITE AND RUTILE

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Indian Standard

METHODS FOR SAMPLING OF ILMENITE AND RUTILE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 June 1967, after the draft finalized by the Methods of Sampling Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The beach sand deposits found mostly in the western coast of India contain large concentrations of heavy minerals like ilmenite and rutile. Recently their production has been increasing considerably and their importance in the export market has also been well recognized. It is therefore, desirable that, due consideration is given to scientific methods of sampling which will help in the proper and objective assessment of the chemical characteristics of the material. Accordingly, the methods of sampling for the concentrated ilmenite and rutile sands as given in this standard are intended to serve this purpose.

0.3 Taking into consideration the views of producers, testing authorities and consumers, the Sectional Committee felt that it should be related to the sampling procedures generally followed in this country for ilmenite and rutile as also for other ores in bulk.

0.4 This standard is one of a series of Indian Standards on sampling of ores and aggregates. Other standards on methods of sampling in this series are:

IS : 1405-1966 Iron ore (*first revision*)
IS : 1449-1961 Manganese ore
IS : 1811-1961 Foundry sands
IS : 1999-1962 Bauxite
IS : 2109-1962 Dolomite, limestone and other allied materials
IS : 2245-1962 Quartzite
IS : 2246-1963 Fluorspar (fluorite)
IS : 4156-1967 Barytes.

0.5 This standard contains clause **7.1** which calls for an agreement between the purchaser and the supplier.

0.6 In reporting the results of test or analysis, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard lays down the procedure to be followed in collecting and preparing samples from a lot in order to determine ore sizes, moisture content and the chemical composition of ilmenite and rutile.

1.1.1 It details the procedures for the sampling of ilmenite and rutile from stock piles and from bags.

1.1.2 This standard also includes a method for reporting the quality of the bulk of the mineral sands (ilmenite and rutile) sampled.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Lot — The quantity of ilmenite or rutile sand indicated to be of the same type and grade offered for inspection at one time. A lot may consist of the whole or a part of the quantity ordered for, or may consist of a part shipment or full shipment.

2.2 Sub-lot — The quantity of ilmenite or rutile in each of the portions into which a lot is divided for the purpose of sampling.

2.3 Increment — The quantity of ilmenite or rutile collected by filling up the sampling scoop to its capacity in a single motion of the scoop or the quantity obtained by mixing up two or more spear or pokeful (as found necessary). The total weight of the increment is about 2 kg only.

2.4 Unit Sample — The quantity of ilmenite or rutile drawn to be representative of a portion (section) of a sub-lot. All the material collected from an auger from a hole of 15 cm diameter encased by a steel casing.

2.5 Gross Sample — The quantity of ilmenite or rutile obtained by mixing together all the increments or unit samples collected from the same sub-lot.

2.6 Laboratory Sample — The quantity of ilmenite or rutile obtained by reducing a gross sample following a specified procedure and intended for laboratory testing.

*Rules for rounding off numerical values (*revised*).

2.7 Composite Sample — The quantity of ilmenite or rutile obtained by mixing together in equal proportions all the laboratory samples obtained from all the gross samples and reduced following the specified procedure for laboratory testing.

2.8 Scoop — A small shovel-like instrument with handle, rectangular in shape with straight sides on 3 sides and open on the fourth side, measuring 175 × 100 × 50 mm and with a capacity to hold approximately 2 kg of the material.

2.9 Spear or Poke — Elongated metallic narrow grooved object with handle-groove length being at least 200 mm, width 5 to 10 mm and depth 3 to 5 mm. The handle is proportionately rounded and its length might vary from 12 to 15 cm. Each spearful of sample weighs from 100 to 200 g.

2.10 Riffle Sample Divider — A Jones Riffle microsplitter with 3 mm wide chutes.

2.11 Sample Division — The process whereby a part of the sample is retained and the remainder rejected.

2.12 Sampling Auger and Casing Pipe — Auger is a screw type of steel drill with a grab at the tip. With a contrivance at the handle the grab may be made to open or close. The grab of the auger when fully open is slightly less in diameter than the internal diameter of the casing pipe. The length of the auger handle varies. A casing pipe is a cylindrical steel pipe open at both ends and with an internal diameter of 15 cm. The length of the casing pipe and the auger used depend on the height of stock pile to be sampled.

3. SAMPLING FROM STOCK PILE

3.0 Stock piles vary greatly in size, particularly in height and hence the collection of representative samples from the stock piles become difficult and costly. For obtaining reliable conclusions, it is recommended that as far as possible ilmenite and rutile be sampled when in motion, that is, during loading or unloading of stock piles (*see* **3.1**). When sampling of stationary stock piles becomes inevitable, sectional sampling (*see* **3.2.1.1**) or trench sampling (*see* **3.2.1.2**) may be used for stock piles up to a maximum height of 1·2 m, after levelling the stock piles to a uniform height. This levelling becomes essential especially in the case of sectional sampling since fine materials like ilmenite and rutile in the sides of the open pits collapse rapidly and flows into the pit.

3.1 Sampling During Loading or Unloading of Stock Pile

3.1.1 *Sub-lots* — The quantity of ilmenite or rutile to be loaded into or unloaded from a stock pile shall be considered as divided into the

number of sub-lots of approximately equal weight, specified below:

Weight of the lot (*tonnes*)	*Number of Sub-lots*
Up to 1000	2
1001 ,, 2000	3
2001 ,, 3500	4
3501 ,, 5000	5

3.1.1.1 While loading or unloading the quantity of ilmenite or rutile constituting a sub-lot, the number of increments specified under 'increment sampling' in Table 1 shall be collected at regular intervals during the whole period of loading or unloading the sub-lot. The increments, each weighing about 2 kg, shall be collected from one or more baskets or bags, as the case may be, filled with ilmenite or rutile.

TABLE 1 NUMBER OF UNIT SAMPLES OR INCREMENTS TO BE COLLECTED FROM A SUB-LOT

(*Clauses* **3.1.1.1, 3.2.1.1, 3.2.1.2** *and* **4.1.1**)

WEIGHT OF THE SUB-LOT (TONNES)	SECTIONAL SAMPLING (NUMBER OF UNIT SAMPLES)	*INCREMENT SAMPLING (NUMBER OF INCREMENTS)
Up to 200	1	10
201 ,, 400	1	20
401 ,, 600	2	30
601 ,, 800	2	45
801 ,, 1000	3	60

*The number of increments refer to only a stock pile of 1·2 m high. For higher stock piles they would be proportionately more.

3.1.2 *Gross Samples* — All increments collected from the same sub-lot shall be mixed together to constitute a gross sample.

3.2 Sampling a Completed Stock Pile

3.2.1 *Sub-lots* — The stock pile is divided into a number of sub-lots of approximately equal weight specified in **3.1.1** by suitably marking the line of demarcation on the surface of the stock pile. The ore surface in the sub-lot shall be levelled before sampling. The sub-lots shall be sampled either by the method of sectional sampling (*see* **3.2.1.1**) or by the method of trench sampling (*see* **3.2.1.2**).

3.2.1.1 *Sectional sampling* — The number of unit samples to be collected from a sub-lot shall depend upon the quantity of the ore in the sub-lot in accordance with Table 1 provided the height of the lot is not

more than 1·2 m. For collecting these unit samples, an equal number of points shall be located at random on the ore surface in the sub-lot.

At every selected point, a unit sample shall be collected by taking the whole section of the ore from top to bottom over an area of a circle of 15 cm diameter. For doing so a sampling auger is used in a casing pipe with an internal diameter of 15 cm. Before using the auger the casing pipe whose length is slightly more than the height of the sub-lot, is pushed vertically down till it touches the bottom of the lot. Then the entire material from the inside of the casing pipe is removed by the auger. The entire quantity of ore removed from the casing pipe forms a unit sample.

3.2.1.2 *Trench sampling* — Each sub-lot shall be trenched in the following manner:

a) The direction and pattern of trenches should be at random without following definite pattern. The pattern should be changed from sub-lot to sub-lot.

b) The trench should extend right up to the bottom of the stack until the ground level is exposed.

c) In addition to the trenches, the sides of the stack should be opened to expose the ore inside the stack at places where the trench does not expose the ore inside.

From the entire cross-section of the exposed sides of the trenches from top to bottom, minimum number of increments, as specified in Table 1 shall be taken. The weight of each increment shall be 2 kg.

3.2.2 *Gross Sample* — All the increments or unit samples collected from the same sub-lot shall be mixed together to constitute a gross sample.

3.3 Thus, a lot will be represented by as many gross samples as the number of sub-lots into which it has been divided. Each gross sample shall be processed further individually in accordance with **5** and **6** and tested in accordance with **7**.

4. SAMPLING FROM BAGS

4.0 Representative samples of ilmenite or rutile may be collected from stacked bags or bags in motion from the stack to boats or bags laying in boats.

4.1 Sub-lot — For the purpose of sampling all the bags in the lot shall be divided into the number of sub-lots of approximately equal weight specified in **3.1.1**.

4.1.1 *Sampling from Stacked Bags or Bags in Motion* — For sampling of ilmenite or rutile in stacked bags, the number of increments specified under increment sampling in Table 1 shall first be noted. For every increment specified 10 bags are selected and from each bag two pokeful or spearful of sample is drawn from opposite ends of the bags or across the diagonals so that the 20 spearful (or pokeful) that go to make up one increment, would weigh about 2 kg. If the bags are piled in regular rows the total number of bags forming the lot are divided into sub-lots as given in **3.1.1** and then the first bag is taken at random and the subsequent bags are taken at regular intervals both horizontally and vertically throughout the sub-lot so that 10 bags are collected for every increment as prescribed in increment sampling of Table 1. From each selected bag two spearful of ore is taken so that the total of 20 spearful forming an increment is nearly 2 kg in weight.

4.2 Gross Sample — All the increments collected from the same sub-lot shall be mixed together to constitute a gross sample.

4.3 Thus, a lot will be represented by as many gross samples as the number of sub-lots into which it has been divided. Each gross sample shall be processed further individually in accordance with **5** and **6** and tested in accordance with **7.**

5. DETERMINATION OF ORE SIZES

5.1 The size distribution of the ilmenite and rutile sand shall normally be estimated as recommended below:

a) Over 0·4 mm,
b) Under 0·4 mm and over 0·25 mm,
c) Under 0·25 mm and over 0·20 mm,
d) Under 0·20 mm and over 0·15 mm,
e) Under 0·15 mm and over 0·10 mm, and
f) Under 0·10 mm.

5.2 Each gross sample shall be screened successively through the relevant sieves and the quantity of ore retained on each of the sieves as also passing through the smallest sieve shall be weighed separately and recorded.

5.2.1 The percentage of each size in a sub-lot (on weight basis) is calculated as follows:

$$\text{Size percentage} = \frac{\text{weight of each size group}}{\text{total weight of the gross sample}} \times 100$$

5.2.2 When the percentage of each size group in the entire lot is required, this shall be calculated as follows:

$$\text{Percentage in each size group} = \frac{a_1 + a_2 + \ldots\ldots}{w_1 + w_2 + \ldots\ldots} \times 100$$

where

a_1, a_2 ..... are the corresponding weights of the respective size group in each of the sub-lots, and

w_1, w_2 ..... are the weights of the different gross samples.

6. REDUCTION OF A GROSS SAMPLE

6.1 Each gross sample shall be reduced separately. The entire quantity of gross sample obtained shall be mixed well and reduced in stages either by coning and quartering or by using Jones Riffle microsplitter till a quantity of 10 kg is obtained. Moisture sample is separated from the quality sample at this stage, 5 kg being quality sample and 5 kg being moisture sample. Now the laboratory sample (quality) and moisture sample are separately reduced till each sample weighs not less than 600 g. The 600 g of quality sample may be further ground to pass through 150-micron IS Sieve.

6.1.1 *Reduction by Coning and Quartering* — The ore shall be well mixed and then scooped into a cone-shaped pile. Care shall be taken to drop each scoopful exactly over the same spot, otherwise the central axis of the cone will be slackened and an uneven distribution of lumps and fines will result. After the cone is formed, it shall be flattened by pressing the top of the cone with the smooth surface of the scoop. Then it is cut into quarters by two lines which intersect at right angles at the centre of the cone. The bulk of the sample is reduced by rejecting any two diagonally opposite quarters.

6.1.2 *Reduction by Using Jones Riffle Microsplitter* — The ore shall be well mixed and poured into the riffle. This process shall be repeated using different size riffles according to the size of the crushed ore.

6.2 The material in each laboratory sample shall be thoroughly mixed and divided into four approximately equal parts. All the four parts are carefully packed and sealed. One sealed sample is taken up for chemical analysis and estimating the main ingredients. The moisture sample is sealed in moistureproof packing, either clean glass bottles, clean tins or plastic bags and sent to the laboratory for moisture determination. One sealed quality sample shall be given to the buyer and one to the seller as the case may be and one sealed sample is kept as a reference sample.

6.2.1 All the samples shall be labelled with full identification, such as the source of the ore, the type of the ore, supplier's name, the lot number and the date of sampling.

7. NUMBER OF TESTS

7.1 All the moisture samples representing a lot shall be tested individually for moisture content. All the laboratory samples for chemical analysis shall be tested individually for important characteristics, for the remaining, a composite sample prepared by mixing equal quantities of the ore from each of the laboratory samples shall be analyzed. The chemical characteristics for which each laboratory sample is to be analyzed individually and also those for which a composite sample is to be tested shall be as given below, unless otherwise agreed to between the purchaser and the supplier:

Chemical Constituents of Ilmenite and Rutile for Which

Laboratory Samples are Analyzed Individually		A Composite Sample is Analyzed	
Ilmenite	Rutile	Ilmenite	Rutile
FeO	Fe	Cr_2O_3	FeO
Fe_2O_3	TiO_2	Al_2O_3	Fe_2O_3
*Monazite	Monazite	MnO	MnO
TiO_2		SiO_2	Cr_2O_3
		CaO	Al_2O_3
		MgO	ZrO_2
		P_2O_5	SiO_2
			CaO
			MgO
			P_2O_5

8. REPORTING

8.1 Reporting of Chemical Composition

8.1.1 For those characteristics, where a composite sample has been tested, only one test result will be available and that result shall be reported as the value of the characteristic for the lot sampled.

8.1.2 When only two laboratory samples have been analyzed individually from a lot, the average of the two available test results shall be reported as the value of the characteristic for the lot sampled. The individual test results shall also be reported to give an indication of the range of variation in quality.

8.1.3 When three or more laboratory samples have been analyzed

*Determined either radiometrically or by microscopic examination.

individually from a lot, the quality of the lot sampled with reference to each of the characteristics shall be reported as follows:

Let $X_1, X_2, X_3 \ldots\ldots X_n$ be the results of analyzing n laboratory samples for a particular characteristic, then,

$$\text{Average } (\bar{X}) = \frac{(X_1 + X_2 + X_3 + \ldots\ldots X_n)}{n}, \text{ and}$$

Range (R) = the difference between the maximum and the minimum values.

The average level of that characteristic in the lot shall be reported as $\bar{X}$.

The limits of variation in the average level of that characteristic in the lot shall be reported as ($\bar{X} \pm hR$) where h is a factor, the value of which depends upon the number of laboratory samples analyzed. The appropriate value of the factor h shall be as given below:

Number of Laboratory Samples Analyzed	*Value of the Factor h*
3	1·30
4	0·72
5	0·51

8.2 Reporting of Moisture Content — The results obtained by testing all the moisture samples representing a lot individually shall be reported in accordance with **8.1.2** or **8.1.3.**

(*Continued from page* 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 1 kg.1 m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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